**Crystallization: Purification of Crude Benzoic Acid and Phenanthrene**

**Introduction**

In this lab the main purpose of this particular lab experiment is to recrystallize benzoic acid and phenanthrene, which was separated in the previous lab experiment. The Benzoic acid would be recrystallized by the use of hexanes while the solvent pair of ethanol and water would recrystallize phenanthrene. The main purpose of the crystallization technique is that its main purpose is the purification of the compounds that were solids at room temperature. Thus, the theory being, that during the process the molecules are deposited from the saturated solution and then according to what shapes they are made of, they fit into crystal lattices. Dissolving a compound in a hot solvent/solvent mixture and then allowing the solution to cool down can do it. Due to the low solubility, thanks to the solvents chosen, it will form crystals in the solutions at lower temperatures.



**Physical Data and Hazards:**

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| --- | --- | --- | --- | --- | --- |
| **Chemical Formula & Name** | **Molecular Weight (g/mol)** | **Melting Point (C)** | **Boiling Point (C)** | **Density (g/cm3)** | **Hazards** |
| *Benzoic Acid* | 122.12 | 122.41 | 249.2 | 1.27 | Irritant |
| *Phenanthrene* | 178.23 | 101 | 340.0 | 1.18 | Moderate health hazard |
| *Water* | 18.02 | 0 | 100 | 1.00 | If inhaled, can be toxic in excess quantities |
| *Hexanes* | 86.18 | 96-94 | 68-69 | 0.6548 | Harmful, flammable, toxic to nature. |
| *Ethanol* | 46.07 | -114 | 78.37 | .7893 | Toxic and flammable |

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

***Safety Precautions***

*Phenanthrene is a moderate health hazard, although gloves and protective clothing should still be worn at all times.*

*Extra care and being precise is needed for this lab because a large volume of CO2 is being produced in this lab as well.*

*Ethanol and hexanes are highly flammable.*

***Wastes***

*Aqueous Wastes:* All ethanol/water filtrates from the recrystallization of phenanthrene in the Aqueous Waste carboy in the main fumehood in the lab.

*Organic Waste:* Place all the hexanes filtrates in the Organic Waste carboy in the main fumehood. *Do NOT place any water or ethanol filtrates in the organic waste.*

*Recovery Jars:* Place your purified compounds in the proper recovery jar in the main fumehood.

**Procedure:**

*Recrystallization of Benzoic Acid*

1. Weigh 0.10g of benzoic acid from previous lab.
   1. Place it in an Erlenmeyer flask
      1. Add 3mL of hexanes and swirl it.
2. Plug in the hot plate, let it pre-heat for about 1 minute of so.
3. Place the Erlenmeyer flask with the benzoic acid and hexanes (solvent-solid mixture) on the hot plate
   1. Heat it up to boiling point.
   2. Intermittently, add 1mL of hexanes and swirl it. Heat it until all the soluble material is dissolved.
4. If solution is colored follow these steps:
   1. Remove solution from hot plate.
   2. Add small amount of pelletized Norite and swirl
   3. Heat up the mixture for up to 5 minutes or until the color disappears.
   4. If the solution is still colored, add a little more of Norite and repeat the process.
5. Now that your color has disappeared, Norite should be removed by decanting the clear liquid into a clean flask and the tiny bit that is left, you can use a Pasteur pipette.
   1. This has to be done while the solution is still warm, do it while its on the hot plate.
6. Remove the benzoic acid from the hot plate and allow it to cool at room temperature. Leave it for 15 minutes, without touching it, you would see crystals being formed.
7. Place it for an ice bath.
8. In order to isolate the crystals, use vacuum filtration. The procedure for vacuum filtration is shown in the previous experiment’s pre-lab procedure.
   1. Use a small amount of chilled hexanes to aid the transfer of solids that are stuck to the walls of the flask.
   2. Let the vacuum filtration go on for 5 minutes.
9. Transfer the crystals into a sample vial, make sure the sample vial is pre-weighed.
10. Allow it to dry.
11. Determine the melting point and weight of the purified benzoic acid.

*Recrystallization of Phenanthrene*

Same procedure as of *Recrystallization of Benzoic acid*, with the following alterations:

1. A solvent pair would be used (ethanol and water).
   1. Due to phenanthrene being soluble in ethanol and insoluble in water.
2. Weigh 0.1g of phenanthrene; dissolve it in 95% ethanol.
3. Add few drops of water until the solution remains cloudy even when its hot.
4. Add couple drops of 95% ethanol to cloudy solution until its clear.
5. Remove the solution from plate and let it cool.
6. After 15 minutes, isolate the crystals by vacuum filtration.
   1. Rinse the crystals on the filter paper with chilled 70% ethanol.
7. Determine the melting point and weight of purified phenanthrene.